M21: ON-LINE SCALE MONITORING DEVICE

By

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Abstract

Developments in evaporator cleaning have accelerated in the past 10 years as a result of an extended period of research into scale formation and scale composition. Chemical cleaning still provides the most cost effective method of cleaning the evaporators.

The paper describes a system that was designed to obtain on-line samples of evaporator scale negating the need to open up hot evaporator vessels for scale collection. This system was successfully implemented in a number of evaporators at a sugar mill. This paper also describes a recent experience in a sugar factory in which the cleaning procedure was slightly modified, resulting in effective removal of intractable scale.

Introduction

During juice evaporation, the heating surface area of sugar mill evaporators becomes increasingly scaled as a result of the deposition of inorganic and organic impurities. With increasing scale deposition the rate that heat can be transferred across the evaporator tubes is diminished and the net effect is a reduction in the capacity to concentrate juice to acceptable brix values. To maintain a nominated cane crushing rate, the No. 1 evaporator (i.e. first effect) calandria pressure will increase over the crushing cycle so as to provide a larger temperature difference across the evaporator set. However, once the low pressure steam valve to the first effect is fully opened, the operation rate for the set will decline due to increased scaling.

As the performance of the evaporator station generally dictates crushing rate and factory stops, improving the production capacity of the evaporator station will have a positive impact on the factory's overall productivity. The production capacity/throughput of the evaporator station can be maximised through a number of strategies including:

- Reducing the rate of scale formation through improved clarification performance. The aim of the clarification stage is to obtain clarified juice with minimum amounts of non-sucrose impurities, as the impurities that are not removed contribute to the amount of scale formed in the evaporators.

- Monitoring the heat transfer coefficient (HTC) of individual evaporators allows the juice operating levels to be controlled and optimised.
- Effective removal of scale. The evaporators' heating surfaces should be devoid of scale so that no nucleation sites are available to accelerate scaling in the subsequent juice processing period and hence impact on HTC. This is achieved through the use of an effective chemical cleaning program. Knowledge on scale composition provides useful information that can be used to identify an effective chemical cleaning program.

Other factors that impact on evaporator station performance are scale composition and intensity associated with cane quality and weather effects. There appears to be a seasonal influence on the rate of scaling and an in-season effect as scaling rates appear to be higher late in the season (Crees *et al.*, 1992).

Many sugar factories are now involved in cogeneration of power in order to increase their product base, and thus become more economically stable by relying less on raw sugar. As a result, reducing the amount of steam used by the factory is necessary which invariably increases the bleeding of vapour from evaporators to heaters and pans. To maintain constant vapour bleeding from an evaporator station, the HTC across the station should be at a high and constant value so that the temperature of bleeding vapour available for heating is adequate.

Cleaning regimes adopted in the industry for evaporator vessels have changed over the years as technology has changed and the sugar industry has moved to continuous crushing. The industry has moved from mechanical brushing to the use of caustic soda (alone), to the use of caustic soda, sulfamic acid and ethylenediaminetetraacetic acid (EDTA) for specific vessels and to the use of special chemical formulations. For a sugar mill to effectively clean an evaporator set throughout the season, a better understanding of the types of scale and distribution through the set is required. This paper describes a system designed to obtain on-line samples of evaporator scales. It also describes how a sugar mill improved chemical cleaning procedures to remove intractable scale.

Evaporator scale monitoring

Sugar factories attain an indication that the amount of scale deposited in an evaporator set is affecting the crushing rate, and must stop and clean the evaporators, from the following observations:

- unacceptably high levels of evaporator supply juice in the tank
- unacceptably high levels of liquor in tanks for evaporators and pans
- low liquor brix
- reduced steam flow rates and hence reduced amount of condensate
- HTC trending towards low values.

Scale sampling probes

Timely analysis of evaporator scale would assist in the identification of the best cleaning formulation for effective scale removal, minimising chemical cleaning costs, downtime and production rate. Obtaining samples of scale during the crushing season is difficult as, even when a mill stops for chemical cleaning, the heat present in the vessels

prevents human access. Consequently, scale samples are normally obtainable during a factory breakdown or at the end of the season. In 1996, Dr Doherty and colleagues at James Cook University, Townsville constructed a mechanical device which enabled scale samples to be collected during cleaning or maintenance stops without the need to wait for the vessels to cool to working temperatures. This device is only suitable for Roberts' evaporator tubes and requires an operator with a measure of skill to effectively remove scales from the tubes. The whole operation takes some time because it requires the manhole of the evaporator to be opened and the operator to wear additional personal protective equipment.

Recently an evaporator probe was designed and constructed to collect scale samples from sugar mill evaporators. The probe was designed so that it could be removed when needed (without requiring the evaporator vessel to be opened) and the scale deposited on the probe surface collected by mechanical means. The surface temperature of the probe was maintained close to that of the calandria within the evaporator vessel by external heating and control. A schematic diagram of the evaporator probe is shown in Figure 1.

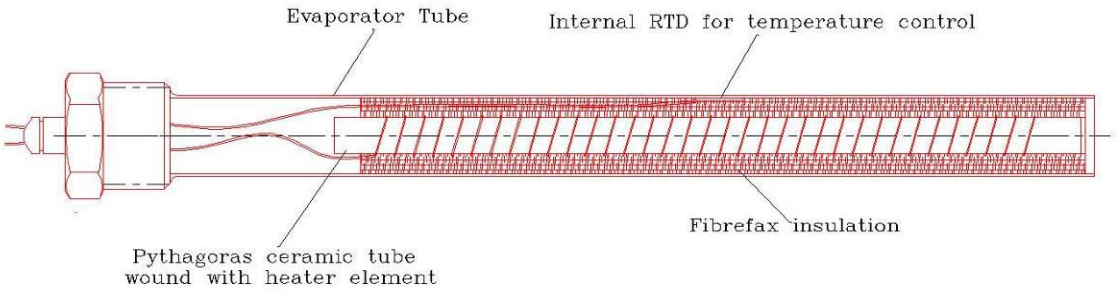

Fig. 1—A cross sectional view of the evaporator scale probe

The evaporator probe was constructed from a 500 mm length of 44.45 mm diameter evaporator tube. This ensures that there is no difference in thermal conductivity between the calandria tubes and the probe. The tube was sealed at one end and was fitted into a machined stainless steel 2" x $1^1/_4$" reducing bush at the other. Pythagoras ceramic tube of 20 mm diameter was then wound with nichrome resistance wire to produce the heater element. The heater element was wrapped with fibrefax insulation and a residence temperature detector (RTD) element was fitted to the external surface of the insulation about half way along the element. The heater element was then inserted into the probe. The heater was powered with 24 V AC and the power to the probe was controlled to keep the probe surface temperature as measured with the internal RTD the same temperature as the LP steam or the vapour in the headspace of the previous vessel. The temperature profile of the probe, monitored over numerous weeks, was within 0.5 to 1 °C of the calandria vapour temperature set point. This is to negate any impact that temperature differences will have on the type of scale deposited (Doherty and Wright, 2004; Gill, 2002; Shames El Din *et al.*, 2005).

Results on scale monitoring during 2008 and 2009 seasons

Analysis of 2008 season scale

The probes were used to monitor scale formation during the 2008 and the 2009 crushing seasons at Tully Sugar Mill. In 2008, scale monitoring was only conducted during the middle of the season. Scale samples were collected from Nos. 1, 2, 4 and 5 evaporators. There was insufficient scale on the No. 3 evaporator's probe. During 2009, scale samples

were collected at the start (Nos. 1 and 4 evaporators), middle (Nos. 3, 4 and 5 evaporators) and end (Nos. 4 and 5 evaporators) of the season. The probes were installed at the start of a crushing cycle and removed before a chemical clean. The duration between cleans at Tully Sugar Mill varied between 13.5 days and 17.5 days.

Rackemann *et al.* (2011), using visual and tactile descriptors, have shown that if the origin of the scale is known, the colour and appearance of scale can be used to qualitatively assess scale composition. Dark coloured scale usually contains mainly hydroxyapatite (HAP) and organic matter, while light coloured scale contains calcium oxalate or is a mixture of calcium oxalate and amorphous silica. Table 1 gives a visual characterisation of the scales collected during the 2008 season. It shows that the scale profile is typical of historical scale composition at Tully Sugar Mill.

Table 1—Visual characterisation of scale samples deposited on probes during middle of the 2008 season

Effect No.	Visual and tactile characterisation	Scale components
1	Dark chocolate scale with a coarse texture. However, on the probe prior to removal, the scale had an almost 'waxy' consistency.	Hydroxyapatite and organic matter. The coarse nature of the scale indicated the presence of silica.
2	Dark chocolate powder with a coarse texture.	Hydroxyapatite, silica and organic matter
4	Light to medium brown scale.	Calcium oxalate and amorphous silica
5	Light coloured scale. This probe contained the largest amount of scale.	Amorphous silica and calcium oxalate

X-ray powder diffraction and X-ray fluorescence were then used to accurately identify the main scale components and their proportions, and the results are summarised in Table 2. Worth noting is the very high proportion of silica in the sample from the No. 1 evaporator and the low oxalate content in the No. 4 evaporator. The proportion of silica is usually at the highest in the final vessels and the proportion of oxalate is usually more than 50% in the No. 4 evaporator (Crees *et al.*, 1992).

Table 2—Scale composition (wt%) of samples collected from the probes at Tully Sugar Mill during the middle of the 2008 crushing season

Component	Effect No.			
	1	2	4	5
Amorphous silica	65	25	52	85
Hydroxyapatite	18	40	21	10
Iron oxide	1	16	2	None
Calcium oxalate	Trace	None	21	6
Organic matter	16	19	4	<1

Analysis of 2009 season scale

At the start of the 2009 season, Tully Sugar Mill experienced reduced scale formation and the only measureable quantities of scale were deposited and collected from the No. 4 evaporator's probe. In the middle of the season there were noticeable increases in scale

intensity and scale samples were collected from Nos. 3, 4 and 5 evaporators' probes. There was a large amount of scale on the Nos. 4 and 5 probes relative to the No. 3 probe. Tully Mill staff observed a large amount of black polymeric material in the No. 3 vessels, which was not readily noticeable on the No. 3 probe. This black polymeric material was collected after chemical cleaning and analysed by micro-elemental analysis and found to have a protein content of 6.6% whereas the protein content on the probe was found to be <1%. This difference between the scale in the probe and that in the No. 3 evaporator is not of any consequence as proteins are readily solubilised in caustic soda solution or in EDTA formulations. At the end of the season, scale samples were also collected from the Nos. 4 and 5 probes.

Table 3 shows that the composition of the scale collected from the probe is similar to the composition of the scale collected from the No. 4 evaporator, further confirming that scale deposition on a probe mimics that on a calandria tube. Scales collected during the middle of the season had similar composition to those collected at the end of season. However, comparing scale samples collected in the seasons of 2008 and 2009 (Tables 2 and 3) shows that the proportions of components vary significantly between the two seasons. While the 2008 season scales contain a very large proportion of silica and a low proportion of calcium oxalate, the trend is reversed in 2009 season. These differences in scale composition are likely due to differences in the cane from which the juices were expressed.

Table 3—Scale composition (wt%) of samples collected from the probes at Tully Sugar Mill at three times during the 2009 season

2009 Season	Early		Middle			Late	
Effect No.	4*	4	3	4	5	4	5
Amorphous silica and silicate	11	9	22	12	13	7	20
Hydroxyapatite	2	3	25	3	1	4	1
Iron oxide	None	1	4	1	1	1	1
Calcium oxalate	72	75	9	80	70	85	72
Organic matter	15	11	40	4	15	3	6

* Scale collected from No. 4 evaporator vessel.

On the basis of the scale composition, the chemical cleaning programs between the two seasons will be different. Scale dissolution studies have previously determined the effects of cleaning chemicals on scale components and these are shown in Table 4 (Doherty, 2000; Rackemann *et al.*, 2011). Caustic soda and sulfamic acid solutions will be effective to attack the scales from the 2008 season, while an EDTA formulation and sulfamic acid will be effective to attack the scale from the 2009 season. It will, however, have been necessary to occasionally treat the evaporators in the 2008 season with EDTA formulation because of the accumulation of oxalate scale. The reduced use of the EDTA formulation would considerably reduce chemical costs, as EDTA is far more expensive than caustic soda or sulfamic acid. This emphasizes the importance of monitoring scale composition in evaporators and the benefits of using an on-line probe device to monitor scale formation. Also, using the evaporator probe to enable visual observations of the scale will provide timely, qualitative information to Mill staff to determine the preferred chemical cleaning program to use during a maintenance stop.

Table 4—Reaction between cleaning agents and scale components

Cleaning agent	Scale component prone to significant dissolution	Scale component prone to partial dissolution
Caustic soda	Organic matter Silicates	Calcium phosphate Hydroxyapatite Amorphous silica Crystalline silica (quartz) Aconitates
EDTA	Oxalates Carbonates Aconitates Sulfates	Calcium phosphate Hydroxyapatite Iron oxide
Acid	Calcium phosphate Hydroxyapatite Carbonates Iron oxide	Sulfates Oxalates Aconitates

Removal of intractable scale at Tableland Sugar Mill

Tableland Mill has a quadruple effect evaporator station. No. 1A evaporator is a falling film plate evaporator and the other vessels (including No. 1B) are Roberts' evaporators. The factory uses direct contact heaters for primary and secondary juice heating. The evaporator supply juice is not preheated before entering the first evaporator vessels and is typically at 97 °C. During the 2011 season, the Mill was unable to effectively remove scale deposited in the Nos. 1B and 3 evaporators using their normal cleaning procedure. As such, scale samples from these evaporators where sent to Queensland University of Technology, Brisbane for analysis in order to determine why the cleaning program used by the Mill was no longer effective.

On the basis of X-ray powder diffraction (XRD) and X-ray fluorescence (XRF) data, No. 1B scale contained 54% hydroxyapatite (Figure 2), 16% amorphous silica, 4% iron oxide and 26% organic matter. The scale from No. 3 evaporator contained 39% hydroxyapatite (Figure 2), 20% amorphous silica, 3% iron oxide and 38% organic matter. The results therefore indicated that scales from No. 1B and No. 3 evaporators contain similar components, though the proportions of these components are different. From the results, it was not evident why the cleaning regime used by the Mill was unsuccessful. XRD and XRF are destructive techniques and only provide the overall chemical information of a scale deposit. No information on the existence of layers or on the composition of the scale surface can be derived using such techniques. Recently East *et al*. (2010) have used scanning electron microscopic methods in combination with X-ray powder diffraction to identify the composition of scale surfaces. Applying a similar approach to the scale samples from Tableland Mill, it was found that a layer of silica (strongly associated with hydroxyapatite) covered the surface of the scales. So it was concluded that the formation of this layer resulted in the ineffective cleaning of the evaporators at Tableland Mill.

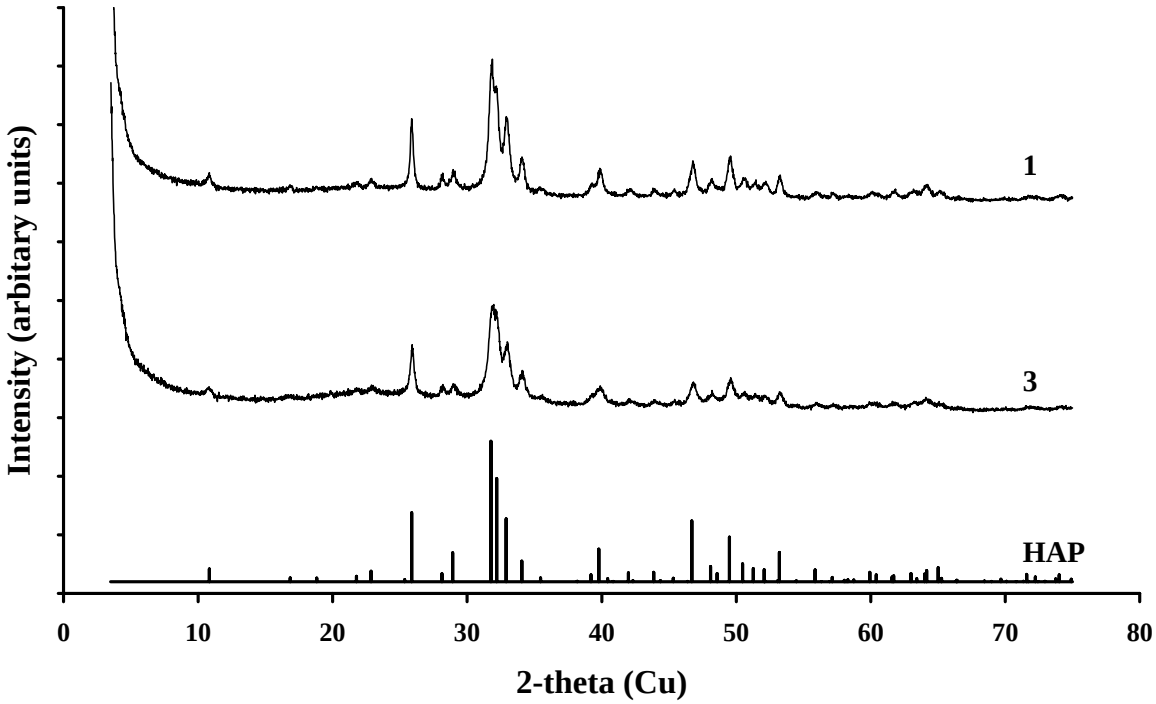

Fig. 2—X-ray powder diffraction pattern of scale from the No. 1B (1) and No. 3 evaporators (3) at Tableland Mill, and the diffraction pattern of hydroxyapatite (HAP)

In the cleaning process at Tableland Mill, sulfamic acid, Rhodine 130 (inhibitor) and caustic soda/EDTA formulation are used in the cleaning of the evaporator set. The normal cleaning process involves adding acid into the No. 4 evaporator and circulating via a dedicated service line to the No. 1A evaporator (vessel level kept constant at 90% to keep plates submerged during boiling) and then sequentially through the set (all Roberts' vessel levels initially set at 65%). The heating time typically employed is ~70 min at a temperature of 80-90 °C maintained under full vacuum and with the boiling action considered as moderate. The operating levels in the Roberts' vessels are ramped down (commencing approximately midway through the acid boil) to a final level of ~45%. This procedure results in very dilute acid solution (<0.5%) made available to attack the scale in the early effects. The acid is then removed, the evaporators rinsed with water, and the caustic soda/EDTA boil is commenced. Caustic solution is added to the Nos. 1 and 2 evaporators, water added to the No. 3 evaporator (acts as a buffer) and EDTA at ~10% concentration added to the No. 4 evaporator. The vessels are boiled separately for 1 h before the set is allowed to recirculate for a further 2 h. The levels in each vessel are the same as for the acid boil.

On the basis of the information obtained on the scale composition the new procedure developed by the Mill involved adding sulfamic acid (and inhibitor) directly to the two No. 1 evaporator vessels to give an initial concentration of ~4%. The vessel levels, heating temperature and boiling time were the same as in the previous cleaning regime, however a more gentle boiling action (lower steam rate) was employed in the No.1 evaporators. The caustic soda/EDTA formulation was also boiled more aggressively (higher steam rate) than was done previously in the 1B vessel. This cleaning protocol was effective in removing the intractable scale because the higher acid concentration used resulted in the dehydration of the silica surface layer thereby exposing the hydroxyapatite component of the scale for solubilisation by the acid. The caustic soda/EDTA formulation was then able to attack the dehydrated silica component, organic matter and iron oxide. Figure 3 shows the HTC of all the evaporator vessels during the period Tableland Sugar Mill used both the old and new

cleaning programs. It shows that the rate of fouling was slower after the use of the modified cleaning program especially in the No. 1 evaporator vessels. This is because of the reduced amount of residual scale on the calandria tubes which provides nucleation sites for scale growth.

It is noted that a number of failed attempts were undertaken to determine a more effective cleaning protocol for the No. 1B vessel trials over several cleaning cycles. An on-line evaporator probe for collecting scale samples may have assisted Tableland Sugar Mill in determining a more effective cleaning protocol in a more timely manner.

Conclusions

An on-line scale sampling device has been designed and implemented in a number of evaporators at a sugar mill. The device represents an additional tool to Mill staff to determine the preferred chemical cleaning program in a timely fashion. Scale samples taken over a number of seasons have shown that the proportion of the scale components can vary significantly from season to season, and so may require slight changes in the chemical cleaning program. Recent experience at another sugar factory found slight modifications to the cleaning procedure were required to allow the effective removal of intractable scale atypical to the factory

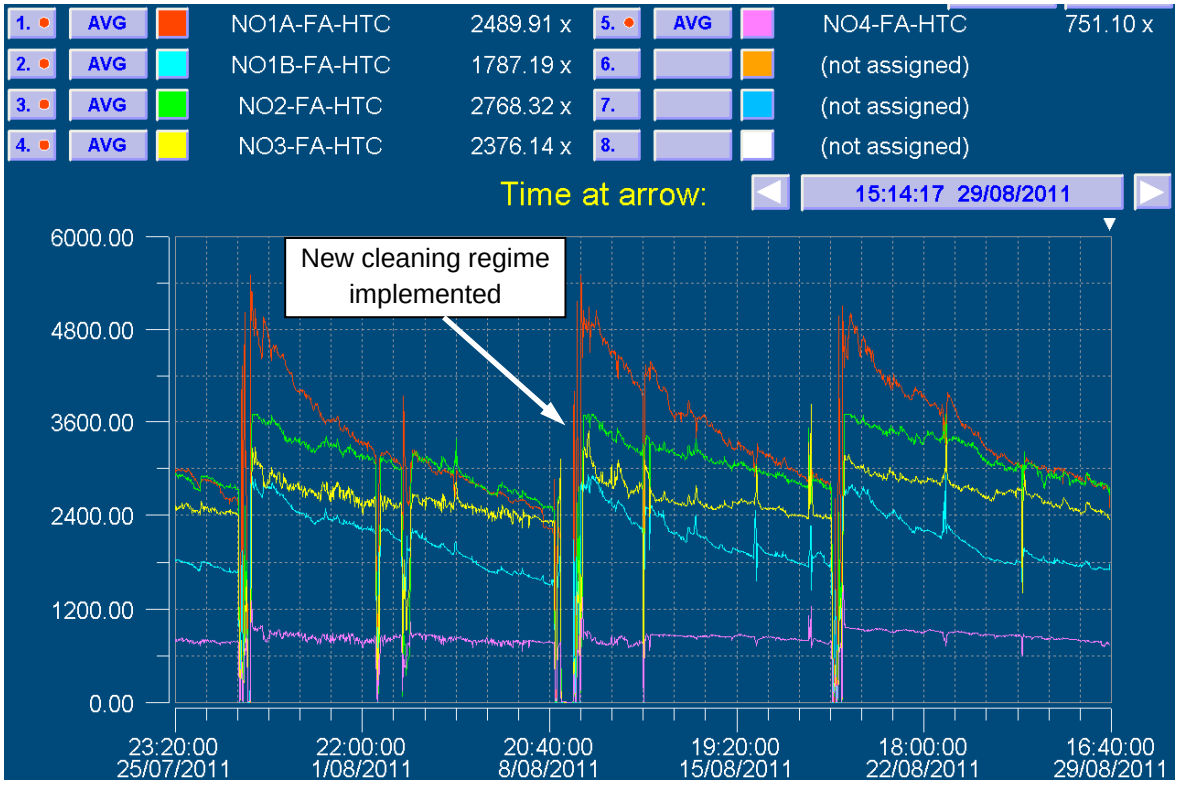

Fig 3.—HTCs' of evaporator vessels at Tableland Sugar Mill during 2011 season when the Mill was experiencing intractable scale

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